

# Structure-Property Relationships in Polyethylene Fusion Bonds

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## Introduction

Polyethylene (PE) is used significantly for a wide range of critical fluid transport applications from water to fossil fuels. The advantages of polyethylene are competitive cost compared to metallic pipe, lower corrosion rates, and toughness. The key to the durability of polyethylene is not only its low reactivity with the environment, but also the microstructure of the material. High performance resins used for piping applications are a bimodal blend of two different polyethylene molecular weights: a medium to high density linear chain and a medium density branched chain (< 10 branches per 1000 monomers). The advantage of the bimodal distribution is that the linear chains crystallize to provide the pipe its strength, while the branched chains resist chain pull-out giving the material exceptional slow crack growth resistance (SCG) [1].

Recently, the nuclear industry has been interested in the application of greater than 1 m diameter pipe for water transportation. While the structure-property relationships for pipe are generally understood, less is known about long term performance of thick walled pipe, particularly the fusion joints used to join these pipe sections. There has been concern that standard test methods for fusion joints do not adequately predict failure time for SCG [2]. In the past few years, the Welding Institute (TWI) in the U.K. has developed a whole pipe creep rupture test that is uniquely suited for long term failure measurements of fusion joints [3]. A challenge for any full pipe based test method is the cost required for evaluating large diameter pipe fusion joints. It would be advantageous to develop a coupon level metrology for conducting design of experiments and sensitivity analysis prior to testing large diameter pipes.

The current research program at National Institute of Standards and Technology (NIST) aims to develop an understanding of the viscoelastic and viscoplastic behaviors for PE resin materials as a function of temperature, pressure, and cooling profiles observed during the formation of butt fusion joints in polyethylene.

## Experimental [4]

Five different PE resins (DOW) were measured for this work. One was a unimodal resin which has a PENT (ASTM F1473) value of ~ 200 hours; while the other four were bimodal resins with PENT times from 5000 hours to 15000 hours. Only the unimodal (DGDB 2480) and one

bimodal (DGDA 2492) will be discussed here. Each resin was compression molded at 160 °C to a 500 µm thickness according to a procedure described in [5]. After pressing, the samples were thermally treated according to the parameters for fusion processing specified in ASME Boiler and Pressure Vessel code case N-755, see Table 1. The resins were exposed to these temperatures and pressures by placing the polyethylene in a metal press, which was heated, under pressure, until temperature equilibrium was reached. The samples were then cooled by passing room temperature (RT) air through the mold platens (3 °C/min). Hereafter, the samples will be denoted by their respective fusion temperature/pressure parameters. In order to bound the behavior, two extremes were examined as a control: a controlled slow cool (1 °C/min) and a quench cool of the samples by passing room temperature (RT) air through the mold platens (3 °C/min). The molding temperature and pressure for these samples was 160 °C and 5.4 MPa.

Table 1. Temperature (T) and pressure (P) exposure for PE resins during melt compression molding.

Resin Name	T (°C)/ P (Pa)	T (°C)/ P (Pa)	T (°C)/ P (Pa)	T (°C)/ P (Pa)
DGDB 2480	204/413	204./620	232/413	232/620
DGDA 2492	204/413	204/620	232/413	232/620

The SCG resistance of polyethylene has been correlated with strain hardening (SH) measurements [5], when those measurements are conducted at 80 °C. The strain hardening modulus is measured as the slope of the stress vs. strain curve after the polyethylene has begun to yield. The current study uses SH measurements of dog bone samples (ASTM D-638), measured at room temperature and at 80 °C, to qualitatively assess SCG resistance of the thermally treated PE. Room temperature SH measurements are used to correlate the indentation measurements, which are conducted at room temperature. The tests were performed at a constant strain rate of 10 mm/min using an electromechanical test machine (Instron). Prior to SH testing, the specimens were allowed to equilibrate at 80 °C in the oven for 30 min. A minimum of three tests was run for each type of specimen. The creep behavior of the resins was measured using instrumented indentation with a 50 µm flat punch indenter over 2000 s under 82 mN of load.

Instrumented indentation is a method to measure elastic (E) and plastic (H) response as well as transient properties (J(t) or G(t)) of small volumes of material. The advantage of indentation is that unlike a fracture energy test, the relevant viscoelastic and viscoplastic properties of a PE fusion bond may be easily mapped spatially without destroying the microstructure. SH at elevated temperatures is required to initiate craze failure within the PE similar to SCG failure, while room temperature measurements are required to relate the SH trends at room temperature to indentation data. The effect of thermal processing on the crystallinity and melting temperature of the resins was characterized using Differential Scanning Calorimetry (DSC). Bulk creep of selected resins was measured under compression.

## Results and Discussion

The SCG growth failure of PE resins is sensitive to the micro-structure of the resin which depends on the polymer as well as the flow and thermal history during processing. For example, Ting and coworkers have shown that bimodal formulations require higher force to initiate failure and have higher ultimate failure strains than unimodal resins [6]. In the case of a fusion bond, which is often used to join pipe segments, the polymer is the same, but the thermal and flow history in the joint region can change local properties so they differ from those of the parent pipe. The thermally affected zone may have altered crystallinity, molecular orientation and, in the case of the bimodal system, changes in composition. The first step of this research was to focus only on thermal processing of the resins to see the impact on SH and local creep behaviors of different conditions, before introducing the effects of flow at the bond line.

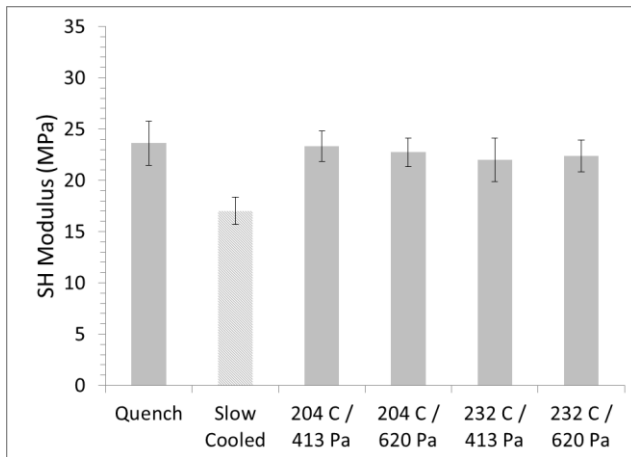


Figure 1. SH Modulus (MPa) for resin 2480 measured at 80 °C as a function of thermal processing during compression molding.

Figure 1 shows the SH modulus at 80 °C for the unimodal resin (2480) as a function of thermal processing. The pressure and temperature of molding have little impact

on the SH, but the cooling rate of the PE plaque changes the SH modulus. Slow cooling the PE reduces the SH modulus indicating a lower resistance to slow crack growth, while quenching the sample increases the SH modulus.

Figure 2 shows the SH modulus at 80 °C for the bimodal resin (2492) as a function of a quench cooled thermal processing. The strain hardening modulus is higher for 2492 compared to that of 2480, which is in agreement with PENT test results of single notch crack growth resistance. In addition, the 2492 resin was less sensitive to cooling rate than the unimodal resin. The SH values measured in this work are also similar to those reported in the literature [6, 7]. Similar to 2480, the cooling rate was the controlling parameter for SCG resistance of the resin.

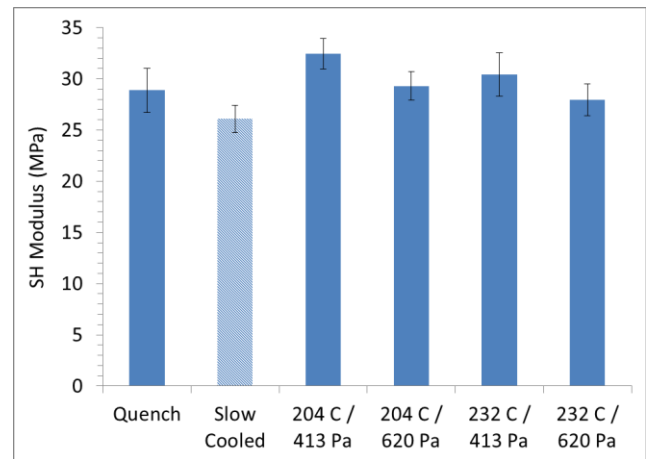


Figure 2. SH Modulus (MPa) for resin 2492 measured at 80 °C as a function of thermal processing during compression molding.

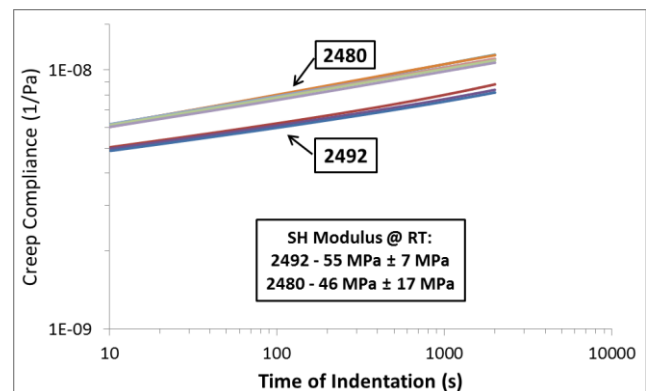


Figure 3. Creep compliance of the quench cooled 2492 and 2480 resins measured using instrumented indentation with a flat punch. The SH modulus under quasi-static tension for each resin, measured at RT, is given in the inset.

Figure 3 shows the creep compliance measured using instrumented indentation for the unimodal and bimodal resins subjected to a quench cooling profile. The nanoindenter

is able to measure differences in the creep behavior of the two resins and indicates the 2480 has higher creep compliance than 2492. This is in agreement with the SH modulus measured at room temperature which is lower than 2492. Although, at room temperature the SH modulus of both resins is considerably higher than at 80 °C. The change in creep compliance as a function of thermal processing and location to the bond line in a model fusion joint will be further described in the presentation.

## Conclusions

SH measurements of PE resins are able to capture the differences between unimodal and bimodal formulations. They are also sensitive to the temperature at which the test is conducted, which reflects the change in polymer failure mode at the different temperatures. It was found that the molding temperature and pressure had less impact on SH modulus than the cooling profile. Instrumented indentation was used to investigate the local creep properties of unimodal and bimodal PE resins. While these measurements do not directly measure the SCG resistance, they do correlate to SH modulus at room temperature. Future work will include spatial investigation of the transient behavior of PE as a function of thermal processing and shear flow in a model fusion joint. These measurements will be correlated to SCG resistance.

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